Chapter NR 219

ANALYTICAL TEST METHODS AND PROCEDURES

Note: A number of the references cited in this chapter are no longer in print. Copies of references which are out-of-print are available at any public library by inter-library loan.

NR 219.01 Purpose. The purpose of this chapter is to establish analytical test methods, preservation procedures, requirements for laboratories, and procedures applicable to effluent limitations for discharges from point sources as authorized by ss. 144.95 and 147.08 (1), Stats.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86; am. Register, June, 1986, No. 366, eff. 7-1-86; am. Register, April, 1988, No. 388, eff. 5-1-88.

NR 219.02 Applicability. (1) The procedures prescribed herein shall, except as provided in s. NR 219.06, be used in the determination of concentrations and quantities of pollutant parameters as required for:

(a) An application submitted to the department for a permit under ch. 147, Stats.

(b) Reports required to be submitted by dischargers in accordance with the conditions of issued permits.

(2) Section NR 219.07 requires that laboratories conducting tests under this chapter be certified, registered, or approved under ch. NR 149, HSS 157 or 165.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. Register, April, 1986, No. 364, eff. 8-28-86; am. (1) (intro.), Register, June, 1986, No. 366, eff. 7-1-86.

NR 219.03 Definitions. As used in this chapter:

(1) "EPA" means the U.S. environmental protection agency.

(2) "Department" means the department of natural resources.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; am. (1), (2), (3) and (4m), Register, January, 1978, No. 265, eff. 2-1-78; r. and recr. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. (1), r. (3) and (4), Register, November, 1992, No. 443, eff. 12-1-92.

NR 219.04 Identification of test procedures. (1) ANALYTICAL TEST PROCEDURES. Parameters or pollutants, for which wastewater analytical methods are approved, are listed together with test procedure descriptions and references in tables A to E. Parameters or pollutants, for which sludge analytical methods are approved, are listed together with test procedure descriptions and references in table EM. The discharge values for the listed parameters shall be determined by one of the standard analytical test procedures identified in a table under this subsection or by an alternate test procedure established under ss. NR 219.05 and 219.06.

(2) PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to E are prescribed in table F. Sludge samples should be preserved at the time of collection by cooling to 4°C. Any per-

son may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

History: Cr. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. Tables B and E, Register, April, 1988, No. 388, eff. 5-1-88; am. Register, November, 1992, No. 443, eff. 12-1-92; am. (1), am. Tables A to F, Register, April, 1994, No. 460, eff. 5-1-94.

NR 219.05 Alternate test procedures. Approvals of alternate test procedures for nationwide use and specific discharges are granted by EPA. An alternate test procedure may only be used if the procedure has been approved by EPA.

Note: The federal requirements for alternate test procedure approval are given in 40 CFR 136.5.

History: Cr. Register, August, 1976, No. 248, eff. 9-1-76; r. and recr. January, 1978, No. 265, eff. 2-1-78; renum. from NR 219.04 and am. Register, June, 1986, No. 366, eff. 7-1-86; r. and recr. Register, November, 1992, No. 443, eff. 12-1-92.

TABLE A

LIST OF APPROVED BIOLOGICAL TEST PROCEDURES FOR WASTEWATER

Move before 219.05

Parameter and Units	Method[1]	EPA	Standard Methods 17th Ed.	USGS
Bacteria:				
1. Coliform (fecal) number per 100 ml	MPN, 5 tube, 3 dilution; or, membrane filter (MF)[2], single step.	p132[3] p124[3]	9221C 9222D	B-0050-85[4]
2. Coliform (fecal) in presence of chlorine number per 100 ml	MPN, 5 tube, 3 dilution; or MF, single step[5]	p132[3] p124[3]	9221C 9222D	
3. Coliform (total) number per 100 ml	MPN, 5 tube, 3 dilution; or, MF[2] single step or two step	p114[3] p108[3]	9221B 9222B	B-0025-85[4]
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or, MF[2] with enrichment.	p114[3] p111[3]	9221B 9222B + B.5c	
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF[2], or Plate count	p136[3] p136[3] p143[3]	9230B 9230C	B-0055-85[4]

Parameter and Units	Method[1]	EPA	Standard Methods 17th Ed.	USGS
Enteroviruses:				
6. Enteroviruses in water, plaque forming units per liter.	Absorption, elution, and organic flocculation, followed by:	Ch. 9[6]	9510G	
	Plaque assay (cell culture infectivity)	Ch. 10[6]	9510G	
	Identification	Ch. 12[6]	9510G	
7. Enteroviruses in sludge, plaque forming units per liter.	Beef extract elution, and organic flocculation, followed by:	Ch. 7[6]	9510F Ch. 9[6]	9510G
	Plaque assay (cell culture infectivity)	Ch. 10[6]	9510G	
	Identification	Ch. 12[6]	9510G	
Mutagenicity:				
8. Mutagenicity (revertants per liter)	Ames test, test strains TA97, TA98, TA100, and TA102.	Note 7		
Acute and Chronic Toxicity:				
9. Toxicity, acute, fresh water organisms, effluent[10]	Daphnia and Ceriodaphnia, 48-h static mortality.	p 56 & 58[8]		
	Fathead minnow, 48-h static mortality, or 48 to 96-h flow-through mortality.	p 60[8]		
10. Toxicity, chronic, fresh water organisms, percent effluent.[10]	Fathead minnow larval survival and growth.	1000.0[9]		
	Fathead minnow embryo-larval survival and teratogenicity.	1001.0[9]		
	Ceriodaphnia survival and reproduction.	1002.0[9]		
	Selenastrum growth.	1003.0[9]		

TABLE A NOTES:

[1] The method used must be specified when results are reported.

[2] A 0.45 μm membrane filter (MF) or other port size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

[3] Bordner, R.H., and J.A. Winter, eds. 'Microbiological Methods for Monitoring the Environment, Water and Wastes', United States Environmental Protection Agency, EPA-600/8-78-017, 1978. Available from ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. Martin Luther King Drive, Cincinnati, Ohio 45268.

[4] Britton, L.J., and P.E. Greeson, eds. '1988 Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples', edited by et al., U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5 chapter A4, Laboratory analysis, 1977. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

[6] Berg, G., R.S. Safferman, D.R. Dahling, D. Berman, and C.J. Hurst, 1984. USEPA Manual of Methods for Virology. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency,

Cincinnati, Ohio. EPA/600/4-84/013. (Chapter 9 revised January 1987; Chapter 10 revised December 1987; Chapter 12 revised May 1988; Chapter 7 revised September 1989).

[7] Williams, L.R., and J.E. Preston, eds. 1983. Interim Procedures for Conducting the Salmonella/Microsomal Mutagenicity Assay (Ames Test). Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Las Vegas, Nevada. EPA/600/4-82/068.

[8] Peltier, W.H., and C.I. Weber, eds. September 1991. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4-90/027.

[9] Weber, C.I., W.H. Peltier, T.J. Norberg-King, W.B. Horning, II, F.A. Kessler, J.R. Menkedick, T.W. Neiheisel, P.A. Lewis, D.J. Klemm, Q.H. Pickering, E.L. Robinson, J.M. Lazorchak, L.J. Wymer, and R.W. Freyberg. 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Surface Waters to Freshwater Organisms, Second Edition, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4-89/001).

[10] Compliance monitoring must be performed in accordance with the specifications in "Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Testing", Wisconsin Department of Natural Resources, May 1992. Available from the Department of Natural Resources Office of Technical Services, P O Box 7921, Madison, WI 53707.

TABLE B

LIST OF APPROVED INORGANIC TEST PROCEDURES FOR WASTEWATER

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
1. Acidity, as $CaCO_3$, mg/L, Electrometric end point or phenolphthalein end point	305.1	2310 B(4a)	B1067-88		
2. Alkalinity, as $CaCO_3$, mg/L; Electrometric or colorimetric:					
Titration to pH 4.5, manual	310.1	2320 B	D1067-88	I-1030-85	973.43[5]
Or automated	310.2				
3. Aluminum-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m]	202.1	3111 D		I-3051-85	
AA furnace,	202.2	3113 B			
Inductively coupled plasma (ICP)[6m]	200.7[7]	3120 B			
Direct current plasma (DCP)[6m], or			D4190-88		Note 36
Colorimetric (Eriochrome cyanine R)		3500-AlD			

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
4. Ammonia (as N), mg/L: Manual distillation[5] (at pH 9.5):					
Followed by	350.2	4500-NH_3B			973.49[5]
Nesslerization,	350.2	4500-NH_3C	D1426-79(A)	I-3520-85	973.46[5]
Titration,	350.2	4500-NH_3E			
Electrode,	350.3	4500-NH_3 F & G	D1426-79(D)		
Automated phenate, or	350.1	4500-NH_3 H	D1426-79(C)	I-4523.85	
Automated electrode					Note 9
5. Antimony - Total[6], ug/L: Digestion[6] followed by:					
AA direct aspiration[6m]	204.1	3111 B			
AA furnace, or	204.2	3113 B			
Inductively coupled plasma[6m]	200.7[7]	3120 B			
6. Arsenic - Total[6], ug/L: Digestion[6] followed by	206.5				
AA (gaseous hydride),		3114	D2972-84(B)	I-3062.85	
AA furnace,	206.2	3113-4d			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
Or, colorimetric (SDDC)	206.4	3500-As	D2972-84(A)	I-3060-85	
7. Barium-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m],	208.1	3111 D		I-3084-85	
AA furnace,	208.2	3113 B			
Inductively coupled plasma[6m], or	200.7[7]	3120 B			
Direct current plasma[6m]					Note 36
8. Beryllium-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration,	210.1	3111 D	D3654-88(A)	I-3095-85	
AA furnace,	210.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
Direct current plasma, or			D4190-88		Note 36
Colorimetric (aluminon)		3500-Be D			
9. Biochemical oxygen demand (BOD5), mg/L:					
Winkler (Azide modifications)		5210		I-1578-78[10]	973.443[5] p. 17[11]
Or electrode method					
10. Boron-Total, mg/L:					
Colorimetric (curcumin),	212.3	4500-B B		I-3112-85	
Inductively coupled plasma,	200.7[7]	3120 B			
or Direct current plasma			D4190-88		Note 36
11. Bromide, mg/L:					
Titrimetric	320.1		D1246-82 (C)(1988)	I-1125-85	p. S44[12]
12. Cadmium-Total[6], mg/L:					
Digestion[6] followed by:					
AA direct aspiration,[6m]	213.1	3111 B or C	D3557-90 (A or B)	I-3135-85 or I-3136-85	974.27[5] p.37[11]
AA furnace,	213.2	3113B			
Inductively coupled plasma[6m]	200.7[7]	3120B		I-1472-85	
Direct current plasma[6m],			D4190-90		Note 36
Voltametry[13], or			D3557-90(C)		
Colorimetric (Dithizone)		3500-Cd D			
13. Calcium-Total[6], mg/L:					
Digestion[6] followed by:					
Atomic absorption,	215.1	3111 B	D511-88(B)	I-3152-85	
Inductively coupled plasma,	200.7[7]	3120 B			
Direct current plasma, or					Note 36
EDTA titration	215.2	3500-Ca D	D511-88(A)		
14. Carbonaceous Biochemical oxygen					

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
demand ($CBOD_5$), mg/L: with nitrification inhibitor[14]		5210 B			
15. Chemical oxygen demand (COD), mg/L: Titrimetric	410.1 410.2 410.3	5220 B	D1252-88	I-3560 or I-3562-85	973.46[5] p.17[11]
Automated and manual Spectrophotometric	410.4			I-3561-85	Notes 15 or16
16. Chloride, mg/L: Titrimetric (silver nitrate) or		4500-Cl B	D512-89(B)	I-1183-85	
(Mercuric nitrate), Colorimetric (ferricyanide), manual or automated	325.3 325.1 or 325.2	4500-Cl C 4500-Cl E	D512-89(A) D512-89(C)	I-1184-85 I-1187-85 I-2187-85	973.51[5]
17. Chlorine - Total residual, mg/L: amperometric, Starch End point direct	330.1 330.3	4500-Cl D 4500-Cl B	D1253-76(A) D1253-76(B) (1985) Part 18.3		
Back Titration either end point[17], or DPD-FAS, Spectrophotometric, DPD; or Electrode	330.2 330.4 330.5	4500-Cl C 4500-Cl F 4500-Cl G			Note 18
18. Chromium VI dissolved, ug/L: 0.45 micron filtration with: Extraction and atomic absorption,	218.4	3111 A		I-1232-85	
Colorimetric (Diphenylcarbazide), or				I-1230-85	307B[19]
Ion Chromatography					218.6[19m]

Parameter, Units & Methods		EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
19.	Chromium-Total[6], mg/L: Digestion[6] (optional extraction) followed by:					
	AA direct aspiration[6m],	218.1	3111 B	D1687-86(D)	I-3236-85	974.24[5]
	AA chelation extraction	218.3	3111 C			
	AA furnace,	218.2	3113B			
	Inductively coupled plasma[6m],	200.7[7]	3120B			
	Direct current plasma[6m], or			D4190-88		Note 36
	Colorimetric (diphenylcarbazide)		3500-Cr D	D1687-84(A)		
20.	Cobalt-Total[6], mg/L: Digestion[6] followed by:					
	AA direct aspiration,	219.1	3111 B (A or B)	D3558-90	I-3239-84	P.37[11]
	AA furnace, or	219.2	3113 B			
	Inductively coupled plasma, or Direct current plasma	200.7[7]	3120 B	D4190-88		Note 36
22.	Copper-Total[6], mg/L: Digestion[6] followed by:					
	AA direct aspiration[6m],	220.1	3111 B or C	D1688-90 (A or B)	I-3271-85 or I-3270-85	974.27[5] p.39[11]
	AA furnace,	220.2	3113 B			
	Inductively coupled plasma[6m]	200.7[7]	3120 B			
	Direct current plasma[6m],			D4190-88		Note 36
	Colorimetric (Neocuproine), or		3500-Cu D or E	D1688-84(88)(A)		
	Bicinchoninate					Note 21
23.	Cyanide - Total, ug/L: Manual distillation with $MgCl_2$		4500-CN-C			
	Followed by: titrimetric,		4500-CN-D			p. 22[11]
	Manual or	335.2	4500-CN-E	D2036-89(A)	I-3300-85	
	Automated[22] spectrophotometric	335.3		D2036-89(A)		

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
24. Cyanide amenable to chlorination, ug/L: Manual distillation with $MgCl_2$ followed by titrimetric, manual or automated spectrophotometric	335.1	4500-CN-G	D2036-89(B)		
25. Fluoride - Total, mg/L: Manual distillation[8]		4500-F-B			
Followed by manual or automated electrode,	340.2	4500-F-C	D1179-88(B)		
				I-4327-85	
SPADNS,	340.1	4500-F-D	D1179-80(A) (1988)		
Or automated complexone	340.3	4500-F-E			
26. Gold Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration	231.1	3111 B			
AA furnace, or	231.2	3113 B			
Direct current plasma					Note 36
27. Hardness - Total as $CaCO_3$, mg/L:					
Automated colorimetric,	130.1				
EDTA titration, (or the sum of Ca and Mg as their respective carbonates by ICP or AA direct aspiration) (See Parameters 13 and 33)	130.2	2340 C	D1126-86 (1990)	I-1338-85	973.52B[5]
28. Hydrogen ion (pH), pH units: Electrometric Measurements or	150.1	4500-H^+ B	D1293-84 (A or B) (1990)	I-1586-85	973.41[5]
Automated Electrode					Note 23

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
29. Iridium - Total[6], ug/L: Digestion[6] followed by:					
AA direct aspiration	235.1	3111 B			
Or AA furnace	235.2				
30. Iron-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m],	236.1	3111 B or C	D1068-84 (C or D)	I-3381-84	973.27[5]
AA furnace,	236.2	3113 B			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
Direct current plasma[6m], or					Note 36
Colorimetric (Phenanthroline)		3500-Fe D	D1068-84(A)		Note 24
31. Kjeldahl nitrogen - Total (as N), mg/L:					
Digestion and distillation	351.3	4500-N org B or C	3590-84(A)		
Followed by titration	351.3	4500-NH_3E	D3590-89(A)		973.46[5]
Nesslerization or	351.3	4500-NH_3C	D3590-89(A)		
Electrode,	351.3	4500-NH_3F or G			
Automated phenate,	351.1	4500-NH_3H		I-4551-78[8]	
Semi-automated block digester,	351.2		D3590-89(B)		
Or potentiometric	351.4		D3590-89(A)		
32. Lead-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m],	239.1	3111 B or C	D3559-85 (A or B)	I-3399-90	974.27[5]
AA furnace,	239.2	3113 B			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
Direct current plasma[6m],			D4190-88		Note 36
Voltametry[13] or			D3559-90(C)		
Colorimetric (Dithizone)		3500-Pb D			

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
33. Magnesium-Total[6], mg/L: Digestion[6] followed by:					
Atomic absorption,	242.1	3111 B	D511-88(B)	I-3447-85	974.27[5]
Inductively coupled plasma,	200.7[7]	3120 B			
Direct current plasma, or					Note 36
Gravimetric		3500-Mg D	D511-77(A)		
34. Manganese-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m],	243.1	3111 B or C	D858-90 (A or B)	I-3454-85	974.27[5]
AA furnace,	243.2	3113 B			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
Direct current plasma[6m],			D4190-88		Note 36
Colorimetric (Persulfate), or		3500-MnD	D858-84(A)(1988)		920.203[5]
Periodate					Note 25
35. Mercury - Total[6], ug/L:					
Cold vapor, manual or automated	245.1 245.2	3112 B	D3223-8	I-3462-85	977.22[5]
36. Molybdenum-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration,	246.1	3111 D		I-3490-85	
AA furnace,	246.2	3113 B			
Inductively coupled plasma,	200.7[7]	3120 B			
or Direct current plasma					Note 36
37. Nickel-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration[6m],	249.1	3111 B or C	D1886-90 (A or B)	I-3499-85	
AA furnace,	249.2	3113 B			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
Direct current plasma[6m], or			D4190-88		Note 36
Colorimetric (Heptoxime)		3500-Ni D			

Parameter, Units & Methods		EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	AA direct aspiration,	255.1	3111B			
	AA furnace, or	255.2				
	Direct current plasma					Note 36
38.	Nitrate (as N), mg/L:					
	Brucine sulfate, or	352.1		D992-71		973.50[5] 419D[19] P. 28[11]
	Nitrate-nitrite N minus Nitrite N (see parameters 39 and 40)					
39.	Nitrate-nitrite (as N), mg/L:					
	Cadmium reduction, manual	353.3	4500-NO_3E	D867-90(B)		
	Or automated,	353.2	4500-NO_3F	D3867-90(A)	I-4545-85	
	or automated hydrazine	353.1	4500-NO_3 H			
40.	Nitrite (as N), mg/L:					
	Spectrophotometric, manual or automated (Diazotization)	354.1	4400-NO_2B	D1254-67	I-4540-85	Note 27
41.	Oil and grease-Total recoverable, mg/L:					
	Gravimetric (extraction)	413.1	5520 B			
42.	Organic carbon - Total (TOC), mg/L:					
	Combustion or oxidation	415.1	5310 B	D2579-85 (A or B)		973.47[5] p. 14[26]
43.	Organic nitrogen (as N), mg/L: Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)					
44.	Orthophosphate (as P), mg/L:					
	Ascorbic acid method, automated	365.1	4500-P F		I-4601-85	973.56[5]

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
Or manual single reagent or	365.2	4500-P E	D515-88(A)		973.55[5]
Manual two reagent	365.3				
45. Osmium - Total[6], ug/L: Digestion[6] followed by:					
AA direct aspiration, or	252.1	3111 D			
AA furnace	252.2				
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification)	360.2	4500-O C	D888-81(C) (1988)	I-1575-78[10]	973.45B[5]
Or electrode	360.1	4500-O G		I-1576-78[10]	
47. Palladium-Total[6], mg/L: Digestion[6] followed by:					
AA direct aspiration,	253.1	3111 B			P.S27[11]
AA furnace, or	253.2				P.S28[11]
Direct current plasma					Note 36
48. Phenols, ug/L:					
Manual distillation[28]	420.1		D1783-80 (A or B)		Note 29
Followed by manual	420.1				
Or automated[22] colorimetric (4AAP)	420.2				Note 29
49. Phosphorus (elemental), mg/L: Gas-Liquid chromatography					Note 30
50. Phosphorus - Total, mg/L:					
Persulfate digestion	365.2	4500-P B,5			973.55[5]
Followed by	365.2 or	4500-P E			
manual or	365.3		D515-88 (A)		
Automated ascorbic acid	365.1	4500-P F		I-4600-85	973.56[5]
Reduction, or semi-automated block digestor	365.4				
51. Platinum-Total[6], mg/L: Digestion[6] followed by:					

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
AA direct aspiration,	255.1	3111 B			
AA furnace, or	255.2				
Direct current plasma					Note 36
52. Potassium - Total[6], mg/L: Digestion[6] followed by:					
Atomic absorption,	258.1	3111 B		I-3620-85	973.53[5]
Inductively coupled plasma,	200.7[7]	3120 B			
Flame photometric, or		3500-K D	D1428-82(A)		
Colorimetric (cobaltinitrate)					317B[19]
53. Residue - total, mg/L: Gravimetric 103-105°C	160.3	2540 B		I-3750-85	
54. Residue - filterable, mg/L: Gravimetric, 180°C	160.1	2540 C		I-1750-85	
55. Residue - nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	2540 D		I-3765-85	
56. Residue - settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	2540 F			
57. Residue - volatile mg/L: Gravimetric, 550°C	160.4	2540 E		I-3753-85	
58. Rhodium - Total[6], ug/L: Digestion[6] followed by:					
AA direct aspiration	265.1	3111 B			
Or AA furnace	265.2				
59. Ruthenium - Total[6] ug/L: Digestion[6] followed by:					

Parameter, Units & Methods	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
AA direct aspiration	267.1	3111 B			
Or AA furnace	267.2				
60. Selenium - Total[6] ug/L: Digestion[6] followed by:					
AA furnace,	270.2	3113 B			
Inductively coupled plasma[6m],	200.7[7]	3120 B			
or AA (gaseous hydride)		3114 B	D3859-88(A)	I-3667-85	
61. Silica - Dissolved, mg/L: 0.45 micron filtration:					
Followed by manual or automated colorimetric (Molybdosilicate), or	370.1	4500-Si D	D859-88(B)	I-1700-85 I-2700-85	
Inductively coupled plasma	200.7				
62. Silver-Total[31], mg/L: Digestion[6] followed by:					
AA direct aspiration,	272.1	3111 B or C		I-3720-85	973.27[5] p 37[11]
AA furnace,	272.2	3113 B			
Colorimetric (Dithizone),					319B[19]
Inductively coupled plasma, or	200.7[7]	3120 B			
Direct current plasma					Note 36
63. Sodium-Total[6], mg/L: Digestion[6] followed by:					
Atomic absorption,	273.1	3111 B		I-3735-85	973.54[4]
Inductively coupled plasma,	200.7[7]	3120 B			
Direct current plasma, or					Note 36
Flame photometric		3500-Na D	D1428-82(A)		
64. Specific conductance, micromhos/cm at 25°C:					

Parameter, Units & Methods		EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	Wheatstone bridge	120.1	2510 B	D1125-82(A)	I-1780-85	973.40[5]
65.	Sulfate (as SO4), mg/L:					
	Automated colorimetric (barium chloroanilate),	375.1				
	Gravimetric, or	375.3	4500-SO_4^{2-}C or D	D516-82(A) (1988)		925.54[5]
	Turbidimetric	375.4		D516-88		426C[32]
66.	Sulfide (as S), mg/L:					
	Titrimetric (iodine) or	376.1	4500-S^{2-}E		I-3840-85	228A[33]
	Colorimetric (methylene blue)	376.2	4500-S^{2-}D			
67.	Sulfite (as SO_3), mg/L:					
	Titrimetric (iodine-iodate)	377.1	4500-$S0_3^{2-}$	D1339-84(C)		
68.	Surfactants, mg/L: Colorimetric (methylene blue)	425.1	5540 C	D2330-88		
69.	Temperature, °C: Thermometric	170.1	2550 B			Note 34
70.	Thallium - Total[6], ug/L: Digestion[6] followed by:					
	AA direct aspiration,	279.1	3111 B			
	AA furnace, or	279.2	3113 B			
	Inductively coupled plasma	200.7[7]				
71.	Tin - Total[6], ug/L: Digestion[6] followed by:					
	AA direct aspiration or	282.1	3111 B		I-3850-78[10]	
	AA furnace	282.2	3113 B			
72.	Titanium-Total[6], mg/L: Digestion[6] followed by:					
	AA direct aspiration,	283.1	3111 D			
	AA furnace, or	283.2	3113 B			

Parameter, Units & Methods		EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]	Other
	Direct current plasma					Note 36
73.	Turbidity, NTU: Nephelometric	180.1	2130 B	D1889-88a	I-3860-85	
74.	Vanadium-Total[6], mg/L: Digestion[6] followed by:					
	AA direct aspiration,	286.1	3111 D			
	AA furnace,	286.2	3113 B			
	Inductively coupled plasma,	200.7[7]	3120 B			
	Direct current plasma, or			D4190-88		Note 36
	Colorimetric (Gallic acid)		3500-V D	D3373-84(A) (1988)		
75.	Zinc-Total[6], mg/L: Digestion[6] followed by:					
	AA direct aspiration[6m],	289.1	3111 B or C	D1691-90 (A or B)	I-3900-85	974.27[5] P.37[11]
	AA furnace,	289.2	3113 B			
	Inductively coupled plasma[6m],	200.7[7]	3120 B			
	Direct current plasma[6m],			D4190-88		Note 36
	Colorimetric (Dithizone), or		3500-Zn E			
	Colorimetric (Zincon)		3500-Zn F			Note 36

TABLE B NOTES:

[1] "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 United States Environmental Protection Agency,Revised March 1983 and 1979 where applicable. Available from National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

[2] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[3] "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1989, unless otherwise stated. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] "Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 15th Edition (1990). Available from The Association of Official Analytical Chemists, 1111 N. 19th Street, Suite 210, Arlington, VA 22209.

[6] For the determination of total metals and total recoverable metals, the sample is not filtered before processing. Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane

filter. A digestion procedure is required to solubilize suspended material and to destroy possible organic metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes", 1979 and 1983. The total metals digestion is a vigorous digestion using nitric acid (4.1.3). The total recoverable metals digestion is a less vigorous digestion using nitric and hydrochloric acids (4.1.4). Use of the graphite furnace technique, inductively coupled plasma, direct current plasma, as well as determinations for certain elements such as arsenic, mercury, selenium, silver, and titanium require a modified digestion and in all cases the method should be consulted for specific instructions and/or cautions. If the digestion included in one of the other approved references is different than the above, the EPA procedure shall be used.

Sample digestion may be omitted for AA (direct aspiration or graphite furnace), direct current plasma, and inductively coupled plasma analyses provided the sample solution to be analyzed meets the following criteria:

(a) has a low COD (<20),

(b) is visibly transparent with a turbidity measurement of 1 NTU or less,

(c) is colorless with no perceptible odor, and

(d) is of one liquid phase and free of particulate or suspended matter following acidification.

[6m] "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1992. Available from the CEM Corporation.

[7] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", is given in Appendix C of 40 CFR 136. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[8] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[9] Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzerII. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[10] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979). Available on inter-library loan.

[11] "American National Standard on Photographic Processing Effluents", April 2, 1975. Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

[12] "Selected Analytical Methods Approved and cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of "Standard Methods for the Examination of Water and Wastewater," from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1981. Available on inter-library loan.

[13] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[14] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required, can the permittee report data obtained using the nitrification inhibitor.

[15] OIC Chemical Oxygen Demand Method. Available from Oceanography International Corporation, 512 West loop, P.O. Box 2980, College Station, TX 77840.

[16] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[17] The back titration method will be used.

[18] ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.

[19] The approved method is that cited in the "Standard Methods for the Examination of Water and Wastewater", 14th Edition, 1976. Available on inter-library loan.

[19m] "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Office of Research and Devel-

opment, Cincinnati, OH 45268, June 1991. Available from the National Technical Information Service (NTIS), order number PB91-231498, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

[20] "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color", NCASI Technical Bulletin No. 253. December, 1971. Available from National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016.

[21] Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[22] After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[23] Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II. Available from Technicon Industrial Systems, Benedict Avenue, Tarrytown, NY 10591.

[24] 1, 10-Phenanthroline Method for Iron, Hach Method 8008, 1980. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[25] Periodate Oxidation Method for Manganese, Method 8034. Hach Handbook of Wastewater Analysis, 1979, pp. 2-113 and 2-117. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[26] "Methods for Analysis of Organic Substances in Water", by D. F. Goerlitz and Eugene Brown: USGS-TWRI, Book 5, Chapter A3, p. 4, 1972. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[27] Nitrite Nitrogen, Hach Method 8507. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[28] Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1+ 9 NaOH.

[29] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 14th Edition. The colorimetric reaction is conducted at a pH of 10.0+ 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure. Available on inter-library loan.

[30] "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

[31] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[32] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 15th Edition. Available on inter-library loan.

[33] The approved method is that cited in "Standard Methods for the Examination of Water and Wastewater", 13th Edition. Available on inter-library loan.

[34] "Water Temperature-Influential Factors, Field Measurement, and Data Presentation", by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS-TWRI Book 1, Chapter D1, 1975. Available from U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[35] Zincon Method of Zinc Method 8009. Hach Handbook for Water Analysis, 1979, pp. 2-231 and 2-333. Available from Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[36] "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.

TABLE C
LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS IN WASTEWATER

		EPA METHOD NUMBER [2,7]					
	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
1.	Acenaphthene	610	625,1625	610	6410 B, 6440 B	D4657-87	
2.	Acenaphthylene	610	625,1625	610	6410 B, 6440 B	D4657-87	
3.	Acrolein	603	[4]624,1624				
4.	Acrylonitrile	603	[4]624,1624	610			
5.	Anthracene	610	625,1625	610	6410 B, 6440 B	D4657-87	
6.	Benzene	602	624,1624		6210 B, 6220 B		
7.	Benzidine		[5]625,1625	605			
8.	Benzo(a)anthracene	610	625,1625	610	6410 B, 6440 B	D4657-87	
9.	Benzo(a)pyrene	610	625,1625	610	6410 B, 6440 B	D4657-87	
10.	Benzo(b)fluoranthene	610	625,1625	610	6410 B, 6440 B	D4657-87	
11.	Benzo(g,h,i)perylene	610	625,1625	610	6410 B, 6440 B	D4657-87	
12.	Benzo(k)fluoranthene	610	625,1625	610	6410 B, 6440 B	D4657-87	
13.	Benzyl chloride						Note 3, p. 130; Note 6, p. S102
14.	Benzyl butyl phthalate	606	625,1625		6410 B		
15.	Bis(2-chloroethoxy) methane	611	625,1625		6410 B		
16.	Bis(2-chloroethyl)ether	611	625,1625		6410 B		
17.	Bis(2-ethylhexyl)phthalate	606	625,1625		6410 B, 6230 B		
18.	Bromodichloromethane	601	624,1624		6410 B, 6230 B		
19.	Bromoform	601	624,1624		6410 B, 6230 B		
20.	Bromomethane	601	624,1624		6410 B, 6230 B		
21.	4-Bromophenylphenyl ether	611	625,1625		6410 B		

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
22.	Carbon tetrachloride	601	624,1624		6230 B, 6410 B		Note 3, p. 130
23.	4-Chloro-3-methylphenol	604	625,1625		6410 B, 6420 B		
24.	Chlorobenzene	601,602	624,1624		6210 B, 6220 B, 6230 B		Note 3, p. 130
25.	Chloroethane	601	624,1624		6210 B, 6230 B		
26.	2-Chloroethylvinyl ether	601	624,1624		6210 B, 6230 B		
27.	Chloroform	601	624,1624		6210 B, 6230 B		Note 3, p. 130
28.	Chloromethane	601	624, 1624		6210 B, 6230 B		
29.	2-Chloronaphthalene	612	625,1625		6410 B		
30.	2-Chlorophenol	604	625,1625		6410 B, 6420 B		
31.	4-Chlorophenylphenyl ether	611	625,1625		6410 B		
32.	Chrysene	610	625,1625	610	6410 B, 6440 B	D4656-87	
33.	Dibenzo(a,h)anthracene	610	625,1625	610	6410 B, 6440 B	D4656-87	
34.	Dibromochloromethane	601	624,1624	610	6410 B, 6440 B	D4656-87	
35.	1,2-Dichlorobenzene	601,602, 612	624,625,1625		6410 B, 6440 B, 6220 B		
36.	1,3-Dichlorobenzene	601,602, 612	624,625,1625		6410 B, 6440 B, 6220 B		
37.	1,4-Dichlorobenzene	601,602, 612	624,625,1625		6410 B, 6440 B, 6220 B		
38.	3,3-Dichlorobenzidine		625,1625	605	6410 B		
39.	Dichlorodifluoromethane	601			6230 B		
40.	1,1-Dichloroethane	601	624,1624		6230 B, 6210 B		
41.	1,2-Dichloroethane	601	624,1624		6230 B, 6210 B		
42.	1,1-Dichloroethene	601	624,1624		6230 B, 6210 B		

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
43.	trans-1,2-Dichloroethene	601	624,1624		6230 B, 6210 B		
44.	2,4-Dichlorophenol	604	625,1625		6420 B, 6410 B		
45.	1,2-Dichloropropane	601	624,1624		6230 B, 6210 B		
46.	cis-1,3 Dichloropropene	601	624,1624		6230 B, 6210 B		
47.	trans-1,3-Dichloropropene	601	624,1624		6230 B, 6210 B		
48.	Diethyl phthalate	606	625,1625		6410 B		
49.	2,4-Dimethlyphenol	604	625,1625		6420 B, 6410 B		
50.	Dimethyl phthalate	606	625,1625		6410 B		
51.	Di-n-butyl phthalate	606	625,1625		6410 B		
52.	Di-n-octyl phthalate	606	625,1625		6410 B		
53.	2,4-Dinitrophenol	604	625,1625		6420 B, 6410 B		
54.	2,4-Dinitrotoluene	609	625,1625		6410 B		
55.	2,6-Dinitrotoluene	609	625,1625		6410 B		
56.	Epichlorohydrin						Note 3, p. 130;Note 6, p. S102
57.	Ethylbenzene	602	624,1624		6220 B, 6210 B		
58.	Fluoranthene	610	625,1625	610	6410 B, 6440 B	D4657-87	
59.	Fluorene	610	625,1625	610	6410 B, 6440 B	D4657-87	
59e.	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin		1613A[8]				
59m.	1,2,3,4,6,7,8-Heptachlorodibenzofuran		1613A[8]				
59t.	1,2,3,4,7,8,9-Heptachlorodibenzofuran		1613A[8]				
60.	Hexachlorobenzene	612	625,1625		6410 B		
61.	Hexachlorobutadiene	612	625,1625		6410 B		
62.	Hexachloro-cyclopentadiene	612	[5]625,1625		6410 B		
62c	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin		1613A[8]				

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
62f.	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin		1613A[8]				
62i.	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin		1613A[8]				
62m.	1,2,3,4,7,8-Hexachlorodibenzofuran		1613A[8]				
62p.	1,2,3,6,7,8-Hexachlorodibenzofuran		1613A[8]				
62s.	1,2,3,7,8,9-Hexachlorodibenzofuran		1613A[8]				
62v.	2,3,4,6,7,8-Hexachlorodibenzofuran		1613A[8]				
63.	Hexachloroethane	612	625,1625		6410 B		
64.	Ideno (1,2-3-cd)pyrene	610	625,1625	610	6410 B, 6440 B	D4657-87	
65.	Isophorone	609	625,1625		6410 B		
66.	Methylene chloride	601	624,1624		6230 B		Note 3, p. 130
67.	2-Methyl-4,6-dinitrophenol	604	625,1625		6420 B, 6410 B		
68.	Naphthalene	610	625,1625	610	6410 B, 6440 B		
69.	Nitrobenzene	609	625,1625		6410 B	D4657-87	
70.	2-Nitrophenol	604	625,1625		6410 B, 6420 B		
71.	4-Nitrophenol	604	625,1625		6410 B, 6420 B		
72.	N-Nitrosodimethylamine	607	[5]625,1625		6410 B		
73.	N-Nitrosodi-n-propylamine	607	625,1625		6410 B		
74.	N-Nitrosodiphenylamine	607	[5]625,1625		6410 B		
74h.	Octachlorodibenzo-p-dioxin		1613A[8]				
74r.	Octachlorodibenzofuran		1613A[8]				
75.	2,2-Oxybis (1-chloropropane)	611	625,1625		6410 B		
76.	PCB-1016	608	625		6410 B		Note 3, p.43
77.	PCB-1221	608	625		6410 B		Note 3, p.43
78.	PCB-1232	608	625		6410 B		Note 3, p.43

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
79.	PCB-1242	608	625		6410 B		Note 3, p.43
80.	PCB-1248	608	625				
81.	PCB-1254	608	625		6410 B		Note 3, p.43
82.	PCB-1260	608	625		6410 B, 6630 B		Note 3, p.43
82e.	1,2,3,7,8-Pentachlorodibenzo-p-dioxin		1613A[8]				
82m.	1,2,3,7,8-Pentachlorodibenzofuran		1613A[8]				
82t.	2,3,4,7,8-Pentachlorodibenzofuran		1613A[8]				
87m.	2,3,7,8-Tetrachlorodibenzofuran		1613A[8]				
83.	Pentachlorophenol	604	625,1625		6410 B, 6630 B		Note 3, p.140
84.	Phenanthrene	610	625,1625	610	6410 B, 6440 B	D4657-87	
85.	Phenol	604	625,1625		6420 B, 6410 B		
86.	Pyrene	610	625,1625	610	6410 B, 6440 B	D4657-87	
87.	2,3,7,8-Tetrachlorodibenzo-p-dioxin			[5a]613, 1613A			
88.	1,1,2,2-Tetrachloroethane	601	624,1624		6230 B, 6210 B		Note 3, p.130
89.	Tetrachloroethene	601	624,1624		6230 B, 6210 B		Note 3, p.130
90.	Toluene	602	624,1624		6210 B, 6220 B		
91.	1,2,4-Trichlorobenzene	612	625,1625		6410 B		Note 3, p.130
92.	1,1,1-Trichloroethane	601	624,1624		6210 B, 6220 B		
93.	1,1,2-Trichloroethane	601	624,1624		6210 B, 6220 B		Note 3, p.130
94.	Trichloroethene	601	624,1624		6210 B, 6230 B		
95.	Trichlorofluoromethane	601	624		6210 B, 6230 B		
96.	2,4,6-Trichlorophenol	604	625,1625		6410 B, 6240 B		

	Parameter[1]	GC	GC/MS	HPLC	Standard Methods[9]	ASTM[10]	Other
97.	Vinyl chloride	601	624,1624		6210 B, 6230 B		

TABLE C NOTES

[1] All parameters are expressed in micrograms per liter (μg/L).

[2] The full text of Methods 601-613, 624, 625, 1624, and 1625, are given in Appendix A of 40 CFR part 136, "Test Procedures for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit." Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

5a 625 Screening only.

[6] "Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of "Standard Methods for the Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1613A, 1624, and 1625 in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

[8] Method 1613 Revision A: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution, HRGC/HRMS, Environmental Protection Agency, Federal Register, page 5098, February 1991. Available from the Superintendent of Documents, US Government Printing Office, Washington, D.C. 20402.

[9] "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 17th Edition, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[10] "1991 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1986. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

TABLE D

LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES IN WASTEWATER[1]

Parameter (micrograms per liter)		Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
1.	Aldrin	GC	608	6630 B & C	D3086-90	Note 3, p.7; Note 4, p.30
		GC/MS	625	6410 B		
2.	Ametryn	GC				Note 3, p. 83; Note 6, p. S68.

Parameter (micrograms per liter)		Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
3.	Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4.	Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5.	Atrazine	GC				Note 3. p. 83; Note 6, p. S68.
6.	Azinphos methyl	GC				Note 3. p. 25; Note 6, p. S51.
7.	Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8.	α-BHC	GC GC/MS	608 [5]625	6630 B & C 6410 B	D3086-90	Note 3, p. 7.
9.	β-BHC	GC GC/MS	608 625	6630 C 6410 B	D3086-90	
10.	δ-BHC	GC GC/MS	608 [5]625	6630 C 6410 B	D3086-90	
11.	γ-BHC (Lindane)	GC GC/MS	608 625	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
12.	Captain	GC		6630 B	D3086-90	Note 3. p. 7.
13.	Carbaryl	TLC				Note 3. p. 94; Note 6. p. S60.
14.	Carbophenothion	GC				Note 4, p. 30; Note 6, p. S73.
15.	Chlordane	GC GC/MS	608 625	6630 B & C	D3086-90	Note 3, p. 7
16.	Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17.	2,4-D	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
18.	4,4'-DDD	GC GC/MS	608 625	6630 B & C 6410 B	D3086-90	Note 3. p. 7; Note 4, p. 30.
19.	4,4'-DDE	GC GC/MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.

Parameter (micrograms per liter)		Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
20.	4,4'-DDT	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410		
21.	Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22.	Demeton-S	GC				Note 3, p. 25; Note 6, p. S51.
23.	Diazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6 p. S51
24.	Dicamba	GC				Note 3, p. 115.
25.	Dichlofenthion	GC				Note 4, p. 30; Note 6, p. S73.
26.	Dichloran	GC		6630 B & C	D3086-90	Note 3, p. 7.
27.	Dicofol	GC				
28.	Dieldrin	GC	608	6630 B & C		Note 3, p. 7; Note 4, p. 30.
		GC/MS	625	6410 B		
29.	Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30.	Disulfoton	GC				Note 3, p. 25; Note 6, p. S51.
31.	Diuron	TLC				Note 3, p. 104; Note 6, p. S64.
32.	Endosulfan I	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
		GC/MS	[5]625	6410 B		
33.	Endosulfan II	GC	608	6630 B & C	D3086-90	Note 3, p. 7.
		GC/MS	[5]625	6410 B		
34.	Endosulfan sulfate	GC	608	6630 C		
		GC/MS	625	6410 B		
35.	Endrin	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
		GC/MS	[5]625	6410 B		
36.	Endrin aldehyde	GC	608	6630 B & C	D3086-90	
		GC/MS	625	6410		

Parameter (micrograms per liter)		Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
37.	Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38.	Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39.	Fenuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40.	Heptachlor	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410 B		
41.	Heptachlor epoxide	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30; Note 6 p. S73
		GC/MS	625	6410 B		
42.	Isodrin	GC				Note 4, p. 30; Note 6, p. S73.
43.	Linuron	TLC				Note 3, p. 104; Note 6, p. S64
44.	Malathion	GC		6630 C		Note 3, p. 25; Note 4, p. 30; Note 6, p. S51
45.	Methiocarb	TLC				Note 3, p. 94; Note 6, p. S60.
46.	Methoxychlor	GC		6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
47.	Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S60.
48.	Mirex	GC		6630 B & C		Note 3, p. 7.
49.	Monuron	TLC				Note 3, p. 104; Note 6, p. S64
50.	Monuron-TCA	TLC				Note 3, p. 104; Note 6, p. S64.
51.	Neburon	TLC				Note 3, p. 104; Note 6, p. S64.

Parameter (micrograms per liter)		Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
52.	Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 30.
53.	Parathion ethyl	GC		6630 B & C	D3086-90	Note 3, p. 25.
54.	PCNB	GC		6630 B & C		Note 3, p. 7.
55.	Perthane	GC			D3086-90	
56.	Prometron	GC				Note 3, p. 83; Note 6, p. S68.
57.	Prometron	GC				Note 3, p. 83; Note 6, p. S68.
58.	Propazine	GC				Note 3, p. 83; Note 6, p. S68.
59.	Propham	TLC				Note 3, p. 104; Note 6, p. S64
60.	Propoxur	TLC				Note 3, p. 94; Note 6, p. S60.
61.	Secbumeton	TLC				Note 3, p. 83; Note 6, p. S68.
62.	Siduron	TLC				Note 3, p. 104; Note 6, p. S64
63.	Simazine	GC				Note 3, p. 83; Note 6, p. S68.
64.	Strobane	GC		6630 B & C		Note 3, p. 7.
65.	Swep	TLC				Note 3, p. 104; Note 6, p. S64.
66.	2,4,5-T	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
67.	2,4,5-TP (Silvex)	GC		6640 B		Note 3, p. 115.
68.	Terbuthylazine	GC				Note 3, p. 83; Note 6, p. S68.
69.	Toxaphene	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30
		GC/MS	625	6410 B		

Parameter (micrograms per liter)	Method	Standard[A] EPA[2,7]	Methods	ASTM[B]	Other
70. Trifluralin	GC		6630 B		Note 3, p. 7.

TABLE D NOTES

[A] "Standard Methods for the Examination of Water and Wastewater", 17th Edition, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[B] "1991 Annual Book of Standards, Water" Section 11, American Society for Testing and Materials, 1980. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table C, where entries are listed by chemical name.

[2] The full text of methods 608 and 625 are given in Appendix A of the Federal Register, October 26, 1984 (Part VIII, 40 CFR part 136), "Test Procedure for Analysis of Organic Pollutants". The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given in Appendix B of 40 CFR part 136, "Definition and Procedure for the Determination of the Method Detection Limit". Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater". U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, 26 W. St. Claire, Cincinnati, Ohio 45268.

[4] "Methods for Analysis of Organic Substances in Water", Book 5, Chapter A3, 1987. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.

[5] The method may be extended to include a(alpha)-BHC, d(delta)-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of "Standard Methods for Examination of Water and Wastewater" (1981). Available from: American Public Health Association, 1015 15th St., N.W., Washington, D.C. 20005.

[7] Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A in 40 CFR part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

TABLE E
LIST OF APPROVED RADIOLOGICAL TEST PROCEDURES FOR WASTEWATER

Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]
1. Alph-Total, pCi per liter	Proportional or Scintillation Counter	900.0	703	D1943-81	pp. 75 and 78[5]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	703	D1943-81	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	703	D1943-81	pp. 75 and 78[5]
4. Beta-Counting error, pCi	Proportional counter	Appendix B	703	D1943-81	p. 79

Parameter and Units	Method	EPA[1]	Standard Methods[2]	ASTM[3]	USGS[4]
5. (a) Radium-Total	Proportional counter	903.0	705	D2460-70	
(b) 226Ra, pCi per liter	Scintillation counter	903.1	706	D3454-79	p. 81

TABLE E NOTES

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/-4-80-032, U.S. Environmental Protection Agency, August 1980.

[2] "Standard Methods for the Examination of Water and Wastewater", 17th Edition, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, 1989. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

[3] "1991 Annual Book of Standards, Water" Section 11, American Society for Testing and Materials, 1980. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

[4] "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976)

[5] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total".

TABLE EM
APPROVED ANALYTICAL METHODS FOR SLUDGE

Parameter	Digestion	Method	Method Number
Metals[1]			
Arsenic	3050A	Inductively Coupled Plasma Emission	6010A
Arsenic	7061A	Gaseous Hydride[2]	7061A
Arsenic	3050A	Graphite Furnace	7060A
Beryllium	3050A	Inductively Coupled Plasma Emission	6010A
Beryllium	3050A	Flame Atomic Absorption	7090
Beryllium	3050A	Graphite Furnace	7091
Cadmium	3050A	Inductively Coupled Plasma Emission	6010A
Cadmium	3050A	Flame Atomic Absorption	7130
Cadmium	3050A	Graphite Furnace	7131A
Chromium	3050A	Inductively Coupled Plasma Emission	6010A
Chromium	3050A	Flame Atomic Absorption	7190
Chromium	3050A	Graphite Furnace	7191
Copper	3050A	Inductively Coupled Plasma Emission	6010A
Copper	3050A	Flame Atomic Absorption	7210
Lead	3050A	Inductively Coupled Plasma Emission	6010A
Lead	3050A	Flame Atomic Absorption	7420
Lead	3050A	Graphite Furnace[3]	7421
Mercury	7471A	Cold Vapor	7471A

TABLE EM
APPROVED ANALYTICAL METHODS FOR SLUDGE

Parameter	Digestion	Method	Method Number
Molybdenum	3050A	Inductively Coupled Plasma Emission	6010A
Molybdenum	3050A	Flame Atomic Absorption	7480
Molybdenum	3050A	Graphite Furnace	7481
Nickel	3050A	Inductively Coupled Plasma Emission	6010A
Nickel	3050A	Flame Atomic Absorption	7520
Selenium	3050A	Inductively Coupled Plasma Emission	6010A
Selenium	7741A	Gaseous Hydride[2]	7741A
Selenium	3050A	Graphite Furnace	7740
Zinc	3050A	Inductively Coupled Plasma Emission	6010A
Zinc	3050A	Flame Atomic Absorption	7950
Biological			
Enteric viruses	NA	Centrifuge Concentration	D 4994-89[4]
Fecal coliform	NA NA	Most Probable Number Membrane Filter	9221 E or 9222 D[5]
Helminth ova	NA	Density Gradient Flotation	[6]
Specific Oxygen Uptake Rate	NA	Respirometer	2710 B[5]
Salmonella	NA	Most Probable Number Selective Media Culture	9260 D.1[5] [7]
Physical			
Solids	NA	Gravimetric	2540 G[5]
Percent Volatiles Solids Reduction	NA	Calculation	[8]

TABLE EM NOTES

1 "Test Methods for Evaluating Solid Waste", SW-846, Office of Solid Waste and Emergency Response, Environmental Protection Agency, November 1986, including December 1987 and July 1992 updates, Washington, DC 20460. Available from the Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250-7954, (202) 783-3238.

2 High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult method 3114, of "Standard Method for the Examination of Water and Wastewater", 17th or 18th edition, for more information.

3 Concentrations of lead in municipal sludge may exceed the working range of Graphite Furnace.

4 "1991 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

5 "Standard Methods for the Examination of Water and Wastewater", 18th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1992. Available from American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

6 "Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1-87-014, Environmental Protection Agency, 1987. Available from the National Technical Information Service, order # PB 88-154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

7 "Determination and Enumeration of *Salmonella* and *Pseudomonas aeruginosa*", Kenner, B.A. and H.A. Clark, J. Water Pollution Control Federation, 46(9):2163-2171, 1994. Available from the Water Environment Federation, 601 Wythe St., Alexandria, VA 22314.

8 "Environmental Regulations and Technology - Control of Pathogens and Bextors in Sewage Sludge", EPA-625/R-92/013, Environmental Protection Agency, Cincinnati, OH, 1992. Available from the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, (703) 487-4650.

TABLE F
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR WASTEWATER

Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
TABLE A - Bacterial Tests:				
1-5.	Bacteria	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$[5]	6 hours
6-7.	Enteroviruses	P,G	Cool, 4°C	24 hours
8.	Mutagenicity	G, Teflon-lined cap	Cool, 4°C	7 days
9-12.	Acute & chronic toxicity	P,G	Cool, 4°C	48 hours
TABLE B - Inorganic Tests:				
1.	Acidity	P,G	Cool, 4°C	14 days
2.	Alkalinity	P,G	Cool, 4°C	14 days
4.	Ammonia	P,G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
9.	Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
11.	Bromide	P,G	None required	28 days
14.	Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
15.	Chemical oxygen demand	P,G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
16.	Chloride	P,G	None required	28 days
17.	Chlorine, total residual	P,G	None required	Analyze immediately
21.	Color	P,G	Cool, 4°C	48 hours
23-24.	Cyanide, total and amenable to chlorination	P,G	Cool, 4°C, NaOH to pH < 12, 0.6g ascorbic acid[5]	14 days[6]
25.	Fluoride	P	None required	28 days
27.	Hardness	P,G	HNO_3 to pH < 2, H_2SO_4 to pH < 2	6 months
28.	Hydrogen ion (pH)	P,G	None required	Analyze immediately

Parameter No./name		Container[1]	Preservation[2,3]	Maximum holding time[4]
31,43.	Kjeldahl and organic nitrogen	P,G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
Metals[7]:				
18.	Chromium VI	P,G	Cool, 4°C	24 hours
35.	Mercury	P,G	HNO_3 to pH < 2	28 days
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29,30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals except chromium VI and mercury		P,G	HNO_3 to pH < 2	6 months
38.	Nitrate	P,G	Cool, 4°C	48 hours
39.	Nitrate-nitrite	P,G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
40.	Nitrite	P,G	Cool, 4°C	48 hours
41.	Oil and grease	G	Cool, 4°C, HCl or H_2SO_4 to pH < 2	28 days
42.	Organic carbon	P,G	Cool, 4°C, HCl or H_2SO_4 to pH < 2	28 days
44.	Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours
46.	Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately
47.	Winkler	G Bottle and top	Fix on site and store in dark	8 hours
48.	Phenols	G only	Cool, 4°C, H_2SO_4 to pH < 2	28 days
49.	Phosphorus (elemental)	G	Cool, 4°C	48 hours
50.	Phosphorus, total	P,G	Cool, 4°C, H_2SO_4 to pH < 2	28 days
53.	Residue, total	P,G	Cool, 4°C	7 days
54.	Residue, Filterable	P,G	Cool, 4°C	7 days
55.	Residue, Nonfilterable (TSS)	P,G	Cool, 4°C	7 days
56.	Residue, Settleable	P,G	Cool, 4°C	48 hours
57.	Residue, Volatile	P,G	Cool, 4°C	7 days
61.	Silica	P	Cool, 4°C	28 days
64.	Specific conductance	P,G	Cool, 4°C	28 days
65.	Sulfate	P,G	Cool, 4°C	28 days
66.	Sulfide	P,G	Cool, 4°C add zinc acetate plus NaOH to pH > 9	7 days
67.	Sulfite	P,G	None required	Analyze immediately
68.	Surfactants	P,G	Cool, 4°C	48 hours

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
69. Temperature	P,G	None required	Analyze immediately
73. Turbidity	P,G	Cool, 4°C	48 hours
TABLE C - Organic Tests[8]			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	14 days
6, 57, 90. Purgeable aromatic	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]HCl to pH<2[9]	14 days
3, 4. Acrolein and acrylonitrile	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], Adjust pH to 4-5[10]	14 days
23, 30, 44, 49, 53, 67, 70, 71, 83,85, 96. Phenols[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
7, 38. Benzidines[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
14 17, 48, 50-52. Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
72-74. Nitrosamines[11,14]	G, Teflon-lined cap	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
76-82. PCBs[11] acrylonitrile	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
54, 55, 65, 69. Nitroaromatics and isophorone[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	7 days until extraction; 40 days after extraction.
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	7 days until extraction; 40 days after extraction.
15, 16, 21, 31, 75. Haloethers[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
29, 35-37, 60-63, 91. Chlorinated hydrocarbons[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction.
59e-59t, 62c-62v, 74h-74r, 82e-82t, 87, 87m. Chlorinated Dioxins and Furans.	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction.
TABLE D - Pesticide Tests:			

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
1-70. Pesticides[11]	G, Teflon-lined cap	Cool, 4°C, pH 5-9[15]	7 days until extraction; 40 days after extraction.
TABLE E - Radiological Tests			
1-5 Alpha, beta, and radium	P,G	HNO_3 to pH < 2	6 months

TABLE F NOTES:

[1] Polyethylene (P) or Glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic)

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting are completed.

[3] When any sample is to be shipped by common carrier or sent through the United States mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table J, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Virus samples can be stored indefinitely at -70°C. Samples used for toxicity tests are to be used for test initiation or for renewal of test solutions within 36 hours of collection as grab samples or after removal from composite samplers. Samples for biological or chemical analysis may be held for longer periods than specified in this table only if the permittee or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator (s. NR 219.05). Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Samples receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O^3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

NR 219.06 Laboratory certification or registration. Bacteriological analyses of groundwater samples, and all radiological analyses shall be performed by the state laboratory of hygiene or at a laboratory certified or approved by the department of health and social services. Other laboratory test results, including effluent toxicity, submitted to the department under this chapter shall be performed by a laboratory certified or registered under ch. NR 149. The following tests are excluded from this requirement:

(1) Temperature,

(2) Turbidity,

(3) Bacteria tests in wastewater effluent,

(4) pH,

(5) Chlorine residual,

(6) Specific conductance,

(7) Physical properties of soils and sludges,

(8) Nutrient tests of soils and sludges,

(9) Flow measurements.

History: Cr. Register, April, 1986, No. 364, eff. 8-28-86; r. and recr. Register, June, 1986, No. 366, eff. 7-1-86; renum. from NR 219.07 r. and recr. Tables A through F, Register, November, 1992, No. 443, eff. 12-1-92; am. (intro.) eff. 7-1-93.